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LOW COST SINTERING OF TITANIUM DIBORIDE FOR ARMOR

October 1988

M. W. VANCE
Aluminum Company of America
Alcoa Technical Center
Alcoa Center, PA 15069

FINAL REPORT

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ABSTRACT

The primary goal of this project was to fabricate lower cost pressureless sintered titanium diboride (TiB_2) armor tiles that would exhibit competitive ballistic performance with tiles formed by hot pressing. This was accomplished by using submicron titanium diboride powders synthesized by a plasma/gas phase reaction process originally developed by Pittsburgh Plate Glass Company. An initial task of Phase I of this effort was to develop and optimize fabrication processes for producing armor tile from plasma TiB_2 powders having various compaction and sintering characteristics. This task was followed by manufacturing sufficient quantities of TiB_2 powder to fabricate 6" x 6" x 1" tiles and a 9" x 9" x 3" tile. From the same lot of powder, eight 6" x 6" x 1" tiles (the machined tile size was later amended to 5.8" x 5.8" x 1") were produced and two of these tiles were used for characterization tests. The results of those tests determined that further tiles required isostatic repressing following initial uniaxial compaction to achieve acceptable properties. The remaining six tiles processed via repressing were delivered to MTL for ballistic tests completing the Phase I effort. Phase II was originally composed of fabricating five (5) 9" x 9" x 3" TiB_2 tiles. Although properties of the Phase I tiles were acceptable, the Phase II effort for producing the 9" x 9" x 3" tiles was not performed, and instead a "best-effort" attempt at manufacturing one large tile to determine process feasibility was implemented. Compaction of the TiB_2 powder into a green tile was accomplished. However, the tile developed excessive crack formation during the sintering process. This same crack formation had been arrested during the fabrication of 6" x 6" x 1" tiles by improving compaction and sintering processes. Also, processes for fabricating 6" x 6" x 1" tiles from high-surface area TiB_2 powders were successfully developed.

During this project, it was observed that to attain low-cost fabrication, machining pressureless sintered TiB_2 tiles to final dimensions should be minimized or eliminated by processing to net-shape dimensions. Non-machined 6" x 6" x 1" tiles were fabricated from the same powder lot to within 1/16" as part of Phase I. However, damage that occurred as a result of repressing required the tiles to be machined to 5.8" x 5.8" x 1.0". Extensive process development will be required to successfully fabricate 9" x 9" x 3" tiles. Additionally, a larger capacity inerted uniaxial press and a continuous charging vacuum furnace would be required to process large quantities of 9" x 9" x 3" tiles to achieve net-shape and low-cost sintering.

FOREWARD

This final report summarizes the development work for the period from 86-04-22 to 88-08-31 on Contract DAAL04-86-R-0041, "Low Cost Sintering of Titanium Diboride for Armor."

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FINAL REPORT
LOW COST SINTERING OF TITANIUM DIBORIDE FOR ARMOR

Contract DAAL04-86-C-0041

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INTRODUCTION

Hot-pressed titanium diboride (TiB_2) armor tile has exhibited superior ballistic performance over other candidate armor materials. However, emphasis in obtaining lower cost armor has resulted in exploring the feasibility of producing pressureless sintered tiles using gas-phase reaction (plasma) synthesized TiB_2 powders. The process for manufacturing these powders was originally developed by Pittsburgh Plate Glass Company (PPG) and later licensed to Alcoa in 1984. Pressureless sintered armor tile manufactured from plasma powders by PPG had exhibited exceptional ballistic performance. However, these tiles were only 4" x 4" x 5/8" thick and were not large enough to determine ballistic performance against kinetic energy threats. This level of testing required 6" x 6" x 1" tiles (minimum size). Therefore, the first phase of this contract [Ref. 1,2] with the Materials Technology Laboratory (MTL) consisted of manufacturing powder and tiles in sufficient quantities and quality by Alcoa to determine ballistic performance of pressureless sintered plasma TiB_2. Successful conclusion of this phase was to lead to a scale-up effort by producing 9" x 9" x 3" thick tiles. All ballistic testing will be conducted by the Army, where the performance of the Alcoa TiB_2 tiles will be compared with that of hot-pressed materials from other sources.

Prior to this contract, process development for fabricating 6" x 6" x 1" armor tiles for in-house ballistic tests was initiated using TiB_2 powders manufactured by PPG [Ref. 3,4] and Alcoa. This resulted in decreasing the experience required to produce tiles using MTL powders. The compaction and sintering procedures for these processes are described in this report. During the powder verification task of Phase I, tiles were compacted using three possible compaction techniques. The first consisted of uniaxial pressing utilizing a hydraulic press to form the basic tile shape. If green densities were not sufficient to obtain the required sintered density and properties, the uniaxial pressed tile underwent a second compaction effort via isostatic press. Finally, if these two procedures failed to produce acceptable tiles, TiB_2 powders were isostatic pressed using a rubber bag/cannister mold. Sintering schedules were varied to optimize properties associated with varying TiB_2 powder lots. After producing sintered 6" x 6" x 1" tiles utilizing one of the aforementioned compaction procedures, materials characterization tests was performed on specimens machined from one of the tiles to determine if the processing developed a ballistic quality armor. These properties included: flexure strength, Young's modulus and microhardness. Microscopic analysis was performed to determine if a fine grain, low porosity microstructure was achieved. Following paragraphs of this report summarize the results required to comply with sections in the Statement of Work and in later amendments mutually agreed on between MTL and Alcoa. These amendments are listed in Appendix A.

II *TASK DESCRIPTION*

The following sections provide a detailed discussion of the work performed during this contract to complete Phase I and to produce a 9" x 9" x 3" tile.

PHASE I: SINTERING PROCESS DEVELOPMENT

The objectives of these tasks were to verify that Alcoa manufactured plasma-produced TiB_2 powders could be fabricated into ceramic armor components, to perform materials characterization studies on specimens taken from as-fabricated plates and to produce ballistic quality 6" x 6" x 1" deliverables.

PHASE I, TASK A: PROCESS VERIFICATION

Prior to initiation of this program, studies were performed on in-house plasma produced TiB_2 powders to determine the optimum compaction and sintering parameters. Because plasma TiB_2 powders are readily sinterable, an important initial stage of achieving acceptable density (>98.5% of maximum theoretical density of 4.52 grams/cc) was to compact and deform waxed agglomerates to an acceptably high green density under the protection of an inert atmosphere followed by sintering them under optimum conditions [Ref. 6]. Overfiring the TiB_2 component would potentially lead to excessive grain growth and microcracking which could decrease mechanical properties. Typical examples of excessive microcracking compared to normal microstructure are shown in the scanning electron microscopic (SEM) photos shown in Figure 1. Potential inability to sinter pyrophoric plasma TiB_2 powders to high densities was traced to exposure of the powders to excessive levels of air or moisture. Although initial studies were often performed successfully on one-inch-diameter bench-scale specimens, compaction and sintering conditions had to be altered significantly to produce 6" x 6" x 1" thick tiles. It became obvious that various powder lots tended to exhibit varying degrees of compaction and sintering. The plan utilized for evaluating the processing characteristics of these different lots of TiB_2 powders is shown in

Figure 2. For some powder lots, uniaxial pressing pressures that would be exerted by the Aeonics Press (75-ton capacity/2600 psi) yielded acceptably high green densities for optimum sintering. A compaction curve for TiB_2 Lot 611745 is shown in Figure 3. For other lots, compaction pressures exceeding the Aeonics Press capabilities were required. Therefore, uniaxial pressed parts whch had been sealed in a rubber bag were repressed at pressures ranging from 10-20 ksi using an isostatic press to attain final green densities that would lead to optimum sintering.

Results of the verification studies conducted on each TiB_2 powder lot to qualify if for fabricating 6" x 6" x1" tiles are discussed in Phase I, Task C. This task on process verification was performed according to C.2.2.1 from the Statement of Work.

PHASE I, TASK B: Powder Processing

Plasma synthesized TiB_2 was prepared using apparatus [Ref. 5] similar to the diagram shown in Figure 4. Primary raw materials were titanium tetrachloride (TCl_4) and boron trichloride (BCl_3) with a chlorinated organic added as a carbon source. The reaction of these gases at very high temperatures produced a submicron (~ 0.5 μ median) particle similar to ones shown in Figure 5. The chemical composition, average particle size and surface area of Alcoa plasma synthesized TiB_2 compared with SDU™ grade carbothermic TiB_2 powders from Advanced Refractory Technologies, Inc. is given below:

Measurement	Plasma Alcoa TiB_2	Carbothermic ART TiB_2 (SDU)
w/o C	0.8	1.3
w/o O	<0.2	1.5
w/o N	-	0.15
w/o Cl	<0.2	-
w/o FE	-	0.02
Surface Area (m^2/gm)	6-13	1.2
Avg. Particle Diameter (μm)	0.5	3.7
Other Elements	*Cu, Fe,Mg, Si <50 ppm each, all others undetetected	

*Analysis by Atomic Emission Spectography

At the completion of this contract, 771.5 lbs. of TiB_2 powder were produced. Although this powder was produced from the same raw materials purchased for this contract, one run usually produced a 60-80 lb. batch. The powder lots produced are summarized in Table I and may represent blends of more than one run prior to the addition of 2.5% w/o wax binder. Variations in surface area were experienced for different powder lots. Decreasing the lot-to-lot variation via blending was beyond the scope of this effort. Completion of the powder production task complied with C.2.2.2 from the Statement of Work.

Phase I, Task C: Fabrication of 6" X 6" X 1" Armor Tiles

The primary objective of this task was to press and sinter 6" x 6" x 1" TiB_2 tiles for characterization and ballistics testing. It was determined during Task A that scaling-up optimized process conditions from one-inch diameter slugs to 6" x 6" x 1" tiles did not always develop acceptable densification for the larger pieces. The data that describes the scale-up of 6" x 6" x 1" tiles as part of the verification of each powder lot produced is given in Table II. Of all the powders evaluated, Lot 611745 (compaction/sintering curve shown in Figure 3) exhibited the most promising sintered density. Where densification was not successfully demonstrated for most of the other powder lots, they were found to have high surface area values (10-13 m^2/gm) compared to PPG powders and to the Lot 611745 powder (6 m^2/gm). After fabricating eight 6" x 6" x 1" tiles from Lot 611745 powder, verification studies continued on the remaining powder lots to develop compaction/sintering processes that would fully densify them. This was accomplished by slowing final sintering ramps to achieve improved densification. The improved firing schedule successfully densified tile No. P-30 for powder Lot 618654. Comparison of firing schedules for tiles from Lots 611745 and 618654 (tile P-30) are given in Tables III and IV, respectively. To minimize pressing defects, new 7.500" x 7.500" dies had been installed prior to this contract to form the best quality tiles possible. Insufficient green density for Aeonics pressed tiles would require repressing of the preformed part at 10-20 ksi. All of these procedures were performed under a protective inert atmosphere. During the verification of Lot 611745, two 6" x 6" x 1" (referring to sintered size) plates were prepared via pressing where one was uniaxially pressed at low pressure (~2600 psi) and a second tile was uniaxially pressed followed by isostatic repressing at 10 ksi to further densify it. After sintering both 6" x 6" x 1" tiles under the same conditions, specimens for flexural strength, microhardness, Young's modulus (sonic method) tests and for microstructural examination were machined. This is described in C.2.3 from the Statement of Work. Material characterization test data comparing properties of the two tiles are given in Table V. A review of the flexure strength, microhardness and Young's modulus values for both tiles indicated that the isostatic repressed plate (P-11) was well within the specifications called out in C.2.3.2, C.2.3.5 and C.2.3.6 from the Statement of Work. The TiB_2 tile (P-10) compacted using the uniaxial press alone did not meet the strength and Young's modulus requirements.

Because tile P-11 had ballistic quality properties, the decision was made to isostatic repress the remainder (6) of the 6" x 6" x 1" tiles for ballistic testing. Because these tiles exhibited increased chipping caused by the rubber bag used for isostatic pressing, the contract was amended [Ref. 7] to allow the size of machined tiles to be decreased from 6" x 6" to 5.8" x 5.8". The specifications for the tile dimensions, tolerances and finish are described in C.2.3.9 (amended) from the Statement of Work. To furnish the lowest cost surface grinding of these tiles, a subcontractor machined the TiB_2 tiles to the revised specifications and their letter of compliance is provided in Appendix B. Additionally, die penetrant inspections were conducted on each of the six tiles using a Magnaflux Corp. ZYGLO™ kit. No cracks were observed to have occurred that had propagated through the tile thickness. However, one small (5/8" long) fold mark caused by isostatic pressing was observed on the top major surface of tile P-15. This mark had not been completely removed by surface grinding. Prior experience using radiography on TiB_2 tiles had failed to show defects that were not detected by this method.

Phase I, Task D: Materials Characterization

The following subtasks describe results of specific characterization tests listed in C.2.3 from the Statement of Work. Some of the data has been previously disclosed in Table V.

Subtask D-1 - Flexure Test Results

The flexure tests [Ref. 8] involved the machining of at least 30 bend bars (MIL STD 1942 MR - "B" size bars) from each of two tiles P-10 (uniaxially pressed) and P-11 (isostatic repressed). The bars were rough machined from the tiles using wire-cut EDM (Electrical Discharge Machining) equipment. This proved to be more cost-effective than cutting TiB_2 with a diamond wheel. At the time of this project, Alcoa did not have EDM equipment and this task was performed by a subcontractor. The bend bars were final ground by a second subcontractor to the final specification. Because the average strength of bars from the P-10 tile (Table V) was found to be below the specification described in C.2.3.2 (amended to 240-360 MPa), the six deliverable tiles were all compacted by repressing. To decrease the probability of green tiles cracking, the isostatic pressure was changed from 20 ksi to 10 ksi.

Subtask D-2 - Weibull Modulus of Specimens from 6" x 6" x 1" Tiles

The Weibull Modulus values were calculated from flexure strength [Ref. 8] values for specimens from P-10 and P-11 tiles and were 10.17 and 11.53, respectively. The Weibull plots comparing these values are shown in Figure 6. The improved Weibull modulus for P-11 may indicate that repressing the green TiB_2 tiles decreases the probability of failure. Completion of this subtask was in compliance with C.2.3.3 from the Statement of Work.

Subtask D-3 - Tile Density and As-Processed Dimensions

The bulk densities (ASTM C20-80A) and as-processed dimensions for the two characterization tiles and six deliverables are given in Table VI. All the repressed tiles had a bulk density of 4.46 to 4.47 gms/cc. This complies with 3.2.3.4 of the Statement of Work. Although the sintered dimensions were all greater than 6" x 6", chips on the tile edges from repressing required surface grinding them to 5.8" x 5.8" x 1". Dimensional tolerances for the deliverables are shown in Figure 7. A hydraulic press with increased capacity for properly compacting the waxed TiB_2 powders would eliminate the need for repressing. The thickness dimensions varied to the greatest degree on a percent basis because of variations in die filling that occurred during pressing. (Plates P-12 through P-16 were all filled using the same die cavity setting.) The variation in sintered dimensions from the mean were as follows:

Length(1)	Width(1)	Thickness(2)
6.148"	6.157"	1.203"
+0.043	+0.017	+0.016
-0.021	-0.015	-0.019

NOTES: (1)For tiles P-12 through P-17

(2)For tiles P-12-through P-16

The fired dimensions and linear shrinkage values for these tiles are listed in Appendix C. The maximum variation in length and width was 0.70% and thickness 1.58% (less than 1/16") for tiles from powder Lot 611745 listed above. The linear shrinkage averaged 17.5% x 17.4% x 17.1% (tiles P-11 through P-17). Shrinkage for the higher surface area TiB_2 powder (618654, tile P-30) was 20.4% x 20.6% x 17.7% and the final tile size was less than 6" x 6". The change in length and width dimensions that was experienced for a given die dimension by changing powders from one lot to another was 0.162" x 0.190" (2.7% x 3.2%). Therefore, variations between various powder lots were significant (1/8 to 3/16"). This was attributed to variations in surface area of the TiB_2 powder produced in the different lots. A blend of powders could have resolved this problem, but this activity was beyond the scope of the project. The original dimensions of the Aeonics Press die were designed to produce oversized tiles. This would allow extra material for machining the tiles to comply with C.2.3.9 (amended). The six 5.8" x 5.8" x 1" TiB_2 tiles submitted to MTL for ballistic testing are shown in Figure 8. Completion of this task complies with the amended version of C.2.3.8.

Subtask D-4 - Microhardness Measurements

The microhardness values reported earlier in Table V indicated that specimens taken from tiles P-10 and P-11 met the specification described in C.2.3.5 from the Statement of Work (Knoop hardness - 2000 ± 250 using a 2000 kg load). These values were 1855 and 1889, respectively. Typical Knoop microhardness values for small specimens representing various powder lots are compared in Table VII.

Subtask D-5 - Young's Modulus

The Young's modulus values for the characterization tiles and deliverables are reported in Table VI. The sintering temperature of these tiles had been increased from 2100 to 2125°C to improve the Young's modulus. These values were calculated using ultrasonic time-of-flight measurements (ASTM procedures E494 and D2845, [Ref. 9,10]. The calculations were based on the assumption that the tiles were composed of isotropic, homogeneous material. The Young's modulus for the deliverables were all better than 80 x 10^6 psi. Therefore, all six tiles conformed to the specification in C2.3.6 from the Statement of Work.

Subtask D-6 - Microstructural Examination

Microstructural specimens were taken at various levels through the thickness of tiles P-10 and P-11 for purposes of determining average grain size, observing presence of the carbide phase which is a characteristic phase in plasma TiB_2 and detecting microcracking and voids. Typical high and low magnification scanning electron microscopic (SEM) photos of tile P-11 are shown in Figure 9. The average grain size was less than 5 µm and there appears to be very little microcrack formation. Pores are attributed to waxed agglomerates that have not deformed properly during pressing and results in voids forming between agglomerates or a wax-rich area that leaves a void during binder removal. A typical defect of this type is shown on the right side of the low magnification SEM, Figure 9, and is approximately 50 µm in diameter. Another source of voids is a clinker that forms during the powder-making process. Defects up to 250 µm in diameter have been observed. The average grain size for specimens taken at various levels through the one-inch thickness from tiles P-10 and P-11 are as follows:

Through-thickness Location	***Average Grain Size (µm) P-10***	***P-11***
Top	3.57	3.73
Middle	3.93	3.58
Middle	3.90	3.82
Bottom	3.55	4.62

This data shows good agreement with an earlier reported relationship between sintering temperature, grain size and bulk density for sintered plasma TiB_2 [Ref. 12]. The grain size of tiles (P-12 through P-17) submitted to MTL may be slightly greater because of an adjustment in sintering temperature.

Phase I Summary

The conclusion of this work resulted in the following accomplishments:

1. The development and optimization of compaction and pressureless sintering processes for obtaining high density 6" x 6" x 1" tiles using TiB_2 powders having a wide range of surface areas..

2. The production of 771.5 lbs. of submicron, high purity TiB_2 powder via plasma synthesis.

3. The fabrication of eight 6" x 6" x 1" TiB_2 tiles from the same powder lot.

4. Characterization of two of the eight tiles. One tile was processed via uniaxial pressing and the other was uniaxially pressed followed by isostatic repressing. The flexure strength, Young's modulus, Knoop hardness values for the repressed tile were all found to be within the values specified in the Statement of Work. The repressed tile exhibited a higher Weibull modulus value than the uniaxial pressed tile.

5. The remaining six tiles were diamond surface ground to 5.8" x 5.8" x 1" and submitted to MTL for ballistic tests. These tiles each had a bulk density of 98.7-98.8% theoretical density and a Young's modulus of approximately 80 x 10^6 psi or better.

6. Tiles repressed and sintered using the same powder lot and processes were net-shaped to within 1/16". A greater variation in dimension occurred for tiles using different powder lots. Blending of lots would be required if large quantities of tiles should be manufactured.

7. Wire-cut EDM is more suitable for cutting dense TiB_2 than diamond sawing. Surface grinding is acceptable for removing thin layers.

8. A uniform, fine-grained microstructure was maintained to ~ 5 μm or less for the relatively large 6" x 6" x 1" tiles and relatively high densities were achieved without excessive grain growth/microcracking.

9" x 9" x 3" Tile Development

At the conclusion of Phase I of this contract, it was agreed [Ref. 13] that fabrication of five 9" x 9" x 3" TiB_2 tiles as described in Phase II should not be performed (C.2.8 of the Statement of Work). This was based on the results of the "best effort" to develop processes and equipment necessary to fabricate a large tile. Therefore, this portion of the technical report will describe tooling and processes that were performed to fabricate the single 9" x 9" x 3" pressureless sintered TiB_2 tile along with final results and recommendations.

Task A - Tooling Development

Isostatic compaction was utilized in lieu of uniaxial pressing for forming the large TiB_2 tile directly from waxed powders. The mold equipment used for fabricating the tile is shown in Figure 10. This was achieved by uniformly filling the rubber bag shown on the right of the photograph with TiB_2 powder. The bag was encased and supported by the aluminum perforated cannister pictured on the left. The as-filled bag was then sealed using a large circular lid and rubber cap which was required to be air and water tight. The filling operation was performed in a large glove box having an argon atmosphere maintained at less than 5 ppm O_2 and H_2O.

Task B - Compaction

The filled mold was pressed at Alcoa Laboratories using an American Autoclave isostatic press (20" in diameter by 72" deep) at a pressure of 10,000 psi. This was performed successfully without cracking or obvious signs of oxidation occurring to the block. The green density was 1.99 gm/cc (44.1% theoretical density) and the dimensions were approximately 13" x 12" x 6".

Task C - Sintering

The tile was transferred into a large graphite box to muffle it from direct exposure to radiation from the vacuum furnace heating elements. It was sintered in vacuum to 2125°C (corrected for window error) for two hours using the schedule given in Table VIII. Ramps in this schedule had been augmented to allow more time for the wax vapor to escape during binder removal and to allow uniform shrinkage to occur throughout the tile during sintering. Furthermore, the soak time was increased to optimize density and the rate of cool-down was decreased to minimize cracking. Cracks observed to occur in smaller tiles were attributed to nonuniform shrinkage due to excessive through-thickness temperature gradients forming during the sintering ramp. The total sintering process for the 9" x 9" x 3" tile required 149 hours to complete.

Examination of the tile after sintering revealed that it had exhibited the crack formation pictured in Figure 10. The final dimensions were approximately 10" x 10 1/2" x 4 3/8". The bulk density of the tile was difficult to estimate because of cracking, but the density of one of the shards was 4.39 gm/cc (97.1% theoretical) indicating that excessive porosity would still have existed even if it was crack-free. A small sample piece sintered as a control during the same firing densified to 98.9% theoretical density.

Summary - Fabrication of the 9" x 9" x 3" Tile

Past experience with sintering crack-free 6" x 6" x 1" tiles revealed that to successfully produce a ballistic quality 9" x 9" x 3" TiB_2 tile, the following modifications to compaction and sintering process should be performed:

1. Compaction procedures should be developed to increase green density.
2. Heating rates during sintering should be decreased to minimize cracking.
3. The soak time should be increased to improve sintered density (>98% theoretical).

To attain net-shape 9" x 9" x 3" tiles by uniaxial pressing, a large capacity press (1000-ton) and special dies would be required. The isostatic bag mold equipment yielded a green tile requiring extensive machining after sintering. This could be minimized by performing some green machining on the isostatic pressed tile prior to sintering if limited quantities would be produced.

CONCLUSIONS

To successfully scale-up from 6" x 6" x 1" to 9" x 9" x 3" pressureless sintered tiles using plasma TiB_2 powder, a significant research effort will be required to develop fabrication processes that will produce a dense, hard, crack-free part. This was evidenced by the experience required to successfully produce quality 6" x 6" x 1" tiles from plasma TiB_2 powders having a wide range of compaction and sintering characteristics. Furthermore, producing pressureless sintered 9" x 9" x 3" tiles will require a large inerted uniaxial press for net-shaping and a vacuum furnace that could be capable of sintering many tiles over a long firing cycle.

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REFERENCES

1. Materials Technology Laboratory Contract DAAL04-86-R-0017, Section C, Statement of Work, Paragraph C.2, Detailed Scope, 1986 January 13.

2. Materials Technology Laboratory Contract DAAL04-86-R-0041, Section C, Statement of Work, Paragraph C.2, Detailed Scope, 1986 April 22.

3. PPG Report, C. B. Holden, "Submicron Products (Process Studies) Density of Green TiB_2 Compacts," 1980 March 12.

4. PPG Report, C. B. Holden, "Submicron Products (Process Studies) - Variability of Green Density in TiB_2 Compacts," 1980 March 10.

5. Alcoa Laboratories Presentation, J. F. Edd, "SiC and TiB_2 Powders via Plasma," The First International Conference on Ceramic Powder Processing Science, ACerS 1987 November 01/04.

6. H. R. Baumgartner and R. A. Steiger, "Sintering and Properties of Titanium Diboride Made From Powder Synthesized in a Plasma-Arc Heater," JACerS, Vol. 67, No. 3, pp. 207-212, 1984 March.

7. Correspondence from P. V. Kelsey , Jr., (Alcoa Laboratories) to P. Wong (MTL), 1986 September 22.

8. Alcoa Laboratories Memorandum, P. R. Bono to M. W. Vance, "RE: Flexure Strength of Alcoa Titanium Diboride," 1987 December 29.

9. Alcoa Laboratories Memorandum, M. P. Jones to M. W. Vance, "RE: Ultrasonic Moduli of TiB_2 Armor Plates," 1987 August 03.

10. Alcoa Laboratories Memorandum, M. P. Jones to M. W. Vance, "RE: Ultrasonic Moduli of TiB_2 Armor Plates," 1987 August 13.

11. Alcoa Laboratories Memorandum, R. C. Stiffler to M. W. Vance, "RE: Ultrasonic Stiffness Determination of Titanium Diboride," 1987 February 13.

12. H. R. Baumgartner, "Mechanical Properties of Densely Sintered High-Purity Titanium Diborides in Molten Aluminum Evnironments," JACerS, Vol. 67, No. 7, pp. 490-497, 1984 July.

13. Correspondence from D. R. Williams (Alcoa Laboratories) to J. J. Walsh (MTL), "RE: Contract DAAL04-86-C-0041, Reduction in Contract Scope," 1988 February 03.

Table 1

List of Powder Lot Numbers and Quantities of TiB_2 Powder Produced for MTL Armor Contract (DAAL04-86-C-0041)

Lot Number	***Original Amount*** [1] (lbs)	***Current Inventory*** [2] (lbs)
611651	84.5	Depleted
611667	104.5	19.5
611745	70.0	Depleted
618654	117.0	71.9
618655	159.0	108.0
RL09.14	76.5 Unwaxed	76.5
RL09.15 and RL09.16	160.0	24.0
Totals	771.5	299.9

NOTES: [1]The overall weight includes 2.5 w/o wax binder unless otherwise noted.

[2]This includes materials currently stored at the Alcoa Laboratories Plasma Fabrication Facility as of 1988 July 12.

TABLE II

Summary of Initial Powder Verification Studies
6" x 6" x 1" and 9" x 9" x 3" Tile

Lot Numbers	Tile Number	Compaction Process Description	Green Density gm/cc/T.D.%	Sintered Density gm/cc/T.D%	Comments
611651	P-1	Uniaxial	2.29/50.6	4.34/95.9	Small specimen sintered to a minimum of 98.5% T.D. 6"x6"x1" tiles failure to achieve adequate density was related to high surface area.
	P-2	Repress @ 20 ksi	2.58/57.1	4.37/96.7	
	P-3	Direct Powder Press @ 20 ksi	2.59/57.3	4.36.96.4	
	P-4 through P-6	Repress @ 20 ksi	2.55/56.4*	4.39/97.1*	Increased sintering time failed to yield adequate density. Tile P-6 was lost during processing.
611667	P-7 through P-9	Repress @ 20 ksi	2.55/56.3*	4.34/96.1*	Increased sintering time and temperature failed to obtain adequate sintered density. This was attributed to high-surface area of the powder.
	P-18	Repress @ 15 ksi	2.43/53.8	4.24/93.9	
	P-22	Direct Powder Press @ 12.5 ksi	2.40/53.1	4.27/94.6	
611745	P-10	Uniaxial	2.58/57.0	4.43/98.0	Improved densification was achieved with low surface area powder. Increased sintering temperature to 2100°C. Repressing required to achieve desired density. (Tile P-11 had ballistic quality properties.) Crack observed in P-11.
	and P-11	Repress @ 20 ksi	2.76/61.0	4.46/98.8	
	P-12 through P-17	Repress @ 10 ksi	Range of Values 2.70-2.73/ 59.6-60.5	Range of Values 4.46-4.47/ 98.7-98.8	Decreased compaction pressure to minimize cracking. Tiles were sintered at 2125°C (corrected). These were delivered to MTL.

NOTE: *Typical values for powders compacted by this process.

TABLE II (Continued)

Summary of Initial Powder Verification Studies
6" x 6" x 1" and 9" x 9" x 3" Tile

Lot Numbers	Tile Number	Compaction Process Description	Green Density gm/cc/T.D.%	Sintered Density gm/cc/T.D%	Comments
618654	P-20	Repress @ 15 ksi	2.61/57.7	4.41/97.6	Small specimens sintered to a minimum of 98.5%T.D. @ 2125°C. This high surface area powder failed to yield satisfactory plate density until sintering ramps were decreased substantially (P-30). However, this plate shrank excessively to below 6" x 6" dimensions.
	P-23	Direct Press Powder @ 12.5 ksi	2.61/57.7	4.43/98.0	
	P-24	Repress @ 10 ksi	2.57/56.8	4.46/98.7	
	P-28	Direct Press Powder @ 10 ksi	2.46/54.4	4.48/98.4	
	P-30	Repress @ 10 ksi	2.57/56.9	4.46/98.7	
618655	P-19	Repress @ 15 ksi	2.54/56.3	4.30/95.0	Small specimens sintered to a minimum of 98.5%T.D. @ 2125°C. Failed to attain acceptable plate density because of high-surface area.
	P-21	Direct Press Powder @12.5 ksi	2.46/54.5	4.24/93.8	
	P-25	Repress @ 10 ksi	2.53/55.9	4.42/97.9	
	P-27	Direct Press Powder @10 ksi	2.49/55.2	4.42/97.8	
Blend of RL-09-15 and RL-09-16	9" x 9" x 3" block	Direct Pressed Powder @10 ksi	1.99/44.1	4.39/97.1	A small specimen sintered to a minimum of 98.5%T.D. @ 2125°C. Green density was very low. Ramps during sintering were substantially decreased for this block over 6" x 6" x 1" tile schedule. Block cracked during sintering after firing for 149 hours.

NOTE: *Typical values for powders compacted by this process

TABLE III

Binder Removal and Sintering Schedule for 6" x 6" x 1" Tiles (Low-Surface Area Powders)

Soak Temperature (°C)	Ramp (°C/Hr)	Soak Time (Hr)	Elapsed Time (Hr)
20 (Start)	-	0.1	0.1
-	30	-	10.1
320[1]	-	1.0	11.1
-	50	-	24.7
1000[2]	-	0.1	24.8
-	102	-	34.2
1950	-	1.0	35.2
-	50	-	38.7
2125[3]	-	1.0	39.7
-	100	-	44.0
1700	-	0.1	44.1
-	195	-	47.7
1000[4]	-	0.1	47.8
-	980	-	48.8
20 (off)	-	Cool freely	

NOTES: (1)Dewax temperature range (0-320°).

(2)Remove thermocouple, initiate Ircon control.

(3)Trim final soak temperature with a Leeds & Northrup optical pyrometer. Temperature includes sight port correction.

(4)Insert thermocouple.

TABLE IV

Binder Removal and Sintering Schedule for 6" x 6" x 1" Tiles (High Surface Area Powders)

Soak Temperature (°C)	Ramp (°C/Hr)	Soak Time (Hr)	Elapsed Time (Hr)
20 (Start)	-	0.1	0.1
-	100	-	3.1
320(1)	-	1.0	4.1
-	200	-	7.5
1000(2)	-	0.1	7.6
-	100	-	14.6
1700	-	0.1	14.7
-	50	-	19.7
1950	-	1.0	20.7
-	50	-	24.2
2125(3)	-	1.0	25.2
-	100	-	29.5
1700	-	0.1	29.6
-	195	-	33.2
1000(4)	-	0.1	33.3
-	980	-	34.3
20 (off)	-	Cool freely	

NOTES: (1)Dewax temperature range (0-320°).

(2)Remove thermocouple, initiate Ircon control.

(3)Trim final soak temperature with a Leeds & Northrup optical pyrometer. Temperature includes sight port correction.

(4)Insert thermocouple.

TABLE V

Comparison of Properties - Uniaxial Pressing versus Isostatic Repressed - 6" x 6" x 1"

Property	Uniaxial Pressing (Tile P-10) [2]	Isostatic Pressing (Tile P-11) [2]	Specification[1]
Green Density	57.0%T.D. 2.58 gm/cc	61.0% T.D. 2.76 gm/cc	N/A[3]
Sintered Density	98.0% T.D. 4.43 gm/cc	98.8% T.D. 4.46 gm/cc	N/A
Final Dimensions (Nonmachined)	6.26" x 6.24" x 1.38"	6.17" x 6.15" x 1.42"	N/A
Flexure Strength [4]	32 x 10^3 (221 MPa)	38 ksi (262 MPa)	34.8 - 52.2 (240-360 MPa)
Standard Dev.	3.4 ksi (23.6 MPa)	3.6 ksi (24.8 MPa)	N/A
Weibull Modulus	10.17	11.53	N/A
Knoop Microhardness[5] (Kg/mm^2)	1855^{+45}_{-95}	1889^{+96}_{-79}	2000 ± 250
Young's Modulus[6] (x 10^6 psi)	73.3	75.9	80.0 ± 5

NOTES:
(1)Specification from the Statement of Work
(2)Powder Batch No. 611745 (MTL)
(3)NA - Not Applicable
(4)MIL-STD 1942 (MR) - B
(5)2000 gram load - ASTM E-384 (Knoop Indenter)
(6)Sonic Measurement

TABLE VI

Data on Deliverable Tiles Fabricated for MTL Contract DAAL04-86-C-0041

Tile Number [1]	Powder Batch Number	Bulk Density gm/cc (T.D. %) Green	Bulk Density gm/cc (T.D. %) Sintered	Young's Modulus [2]	Dimension As-Fired (inches)	Comments
P-10	S-611745	2.58 (57.0)	4.43 (98.0)	73.3[3]	6.26x6.24x1.38	Uniaxially pressed only. Bend bars were machined from this tile.
P-11	S-611745	2.76 (61.0)	4.46 (98.8)	75.9[3]	6.17x6.15x1.42	Isostatic repressed . Bend bars were machined from this tile.
P-12	S-611745	2.70 (59.7)	4.46 (98.7)	80.2[3]	6.19x6.17x1.22	Submitted to MTL.
P-13	S-611745	2.71 (60.0)	4.46 (98.7)	80.4[3]	6.17x6.17x1.20	Submtted to MTL.
P-14	S-611745	2.72 (60.2)	4.46 (98.7)	80.8[4]	6.13x6.14x1.21	Submitted to MTL
P-15	S-611745	2.70 (59.6)	4.46 (98.7)	81.0[4]	6.13x6.14x1.18	Submitted to MTL.
P-16	S-611745	2.73 (60.4)	4.47 (98.8)	80.5[4]	6.13x6.15x1.20	Submitted to MTL.
P-17	S-611745	2.73 (60.5)	4.46 (98.7)	80.9[4]	6.13x6.16x1.14	Submitted to MTL.

NOTES:
(1)Alcoa Laboratories Notebook No. 22877
(2)x 10^6 psi (Specification $80 \pm 5 \times 10^6$ psi)
3)Reference 9
(4)Reference 10

TABLE VII

Knoop Hardness Values for Sintered TiB_2 Powders

Manufacturer	Knoop Hardness (2000 gm Load)[1] Specimen 1	Specimen 2	Bulk Density[5] (%T.D.)
PPG[2] (Lot 2252/2464)	1890	1921	97.2
PPG[2] (Lot 2793)	1953	1953	93.0
Alcoa[3] (Lot 591160)	2050	2050	98.0
Alcoa[4] (Lot 618654)	2198	2020	98.4
Alcoa[4] (Lot 611659	2020	2020	98.1
Alcoa[4] (Lot 611667)	1525	1669	98.1
Alcoa[4] (Lot 611745)	2160	2122	98.3

NOTES:

[1]MTL Specifications - 2000 ± 250

[2]PPG (Pittsburgh Plate Glass Corp.) TiB_2 powder synthesized powders

[3]Powders produced by Alcoa for internal use

[4]Powders produced for the MTL contract DAAL04-86-R-0041

[5]All specimens were small (1/2" diameter) and were sintered at 2100°C (corrected)

TABLE VIII

Binder Removal and Sintering Schedule for 9" x 9" x 3" Tile

Soak Temperature (°C)	Ramp (°C/Hr)	Soak Time (Hr)	Elapsed Time (Hr)
20 (Start)	-	0.1	0.1
-	20	-	15.1
320 (1)	-	3.0	18.1
-	20	-	52.1
1000 (2)	-	0.1	52.2
-	20	-	87.2
1700(3)	-	1.0	88.2
-	20	-	100.7
1950 (3)	-	1.0	101.7
-	20	-	110.5
2125(4)	-	2.0	112.5
-	20	-	133.8
1700	-	0.1	133.9
-	50	-	147.9
1000(5)	-	0.1	148.0
-	980	-	149.0
20 (off)	-	Cool freely	

NOTES: (1)Dewax temperature range (0-320°C)

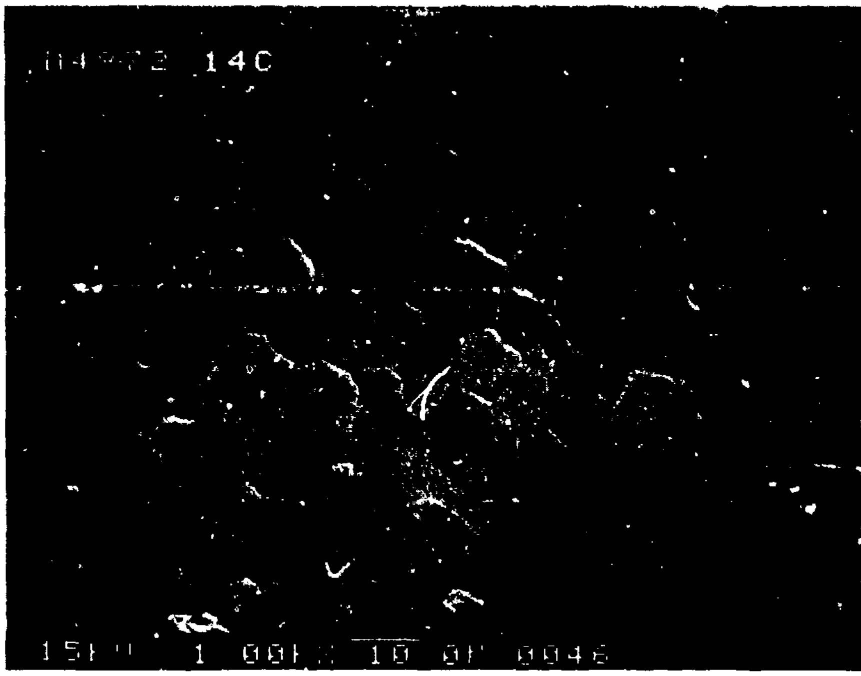
(2)Remove thermocouple, initiate Ircon control

(3)Equilibrium hold

(4)Maximum soak temperature was trimmed using a Leeds and Northrup optical pyrometer. Temperature includes sight port correction.

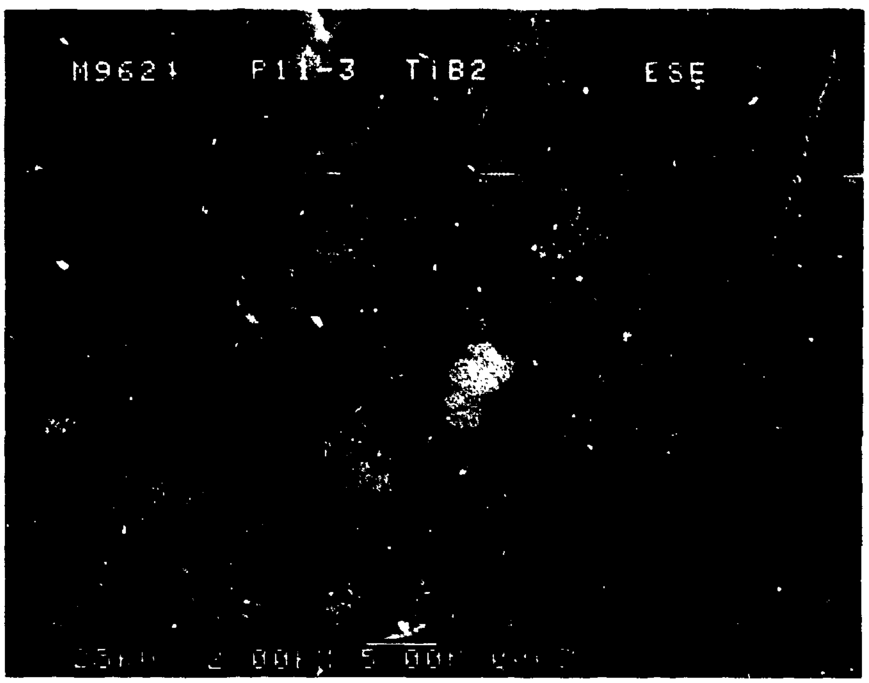
(5)Insert thermocouple

GA 23696.2



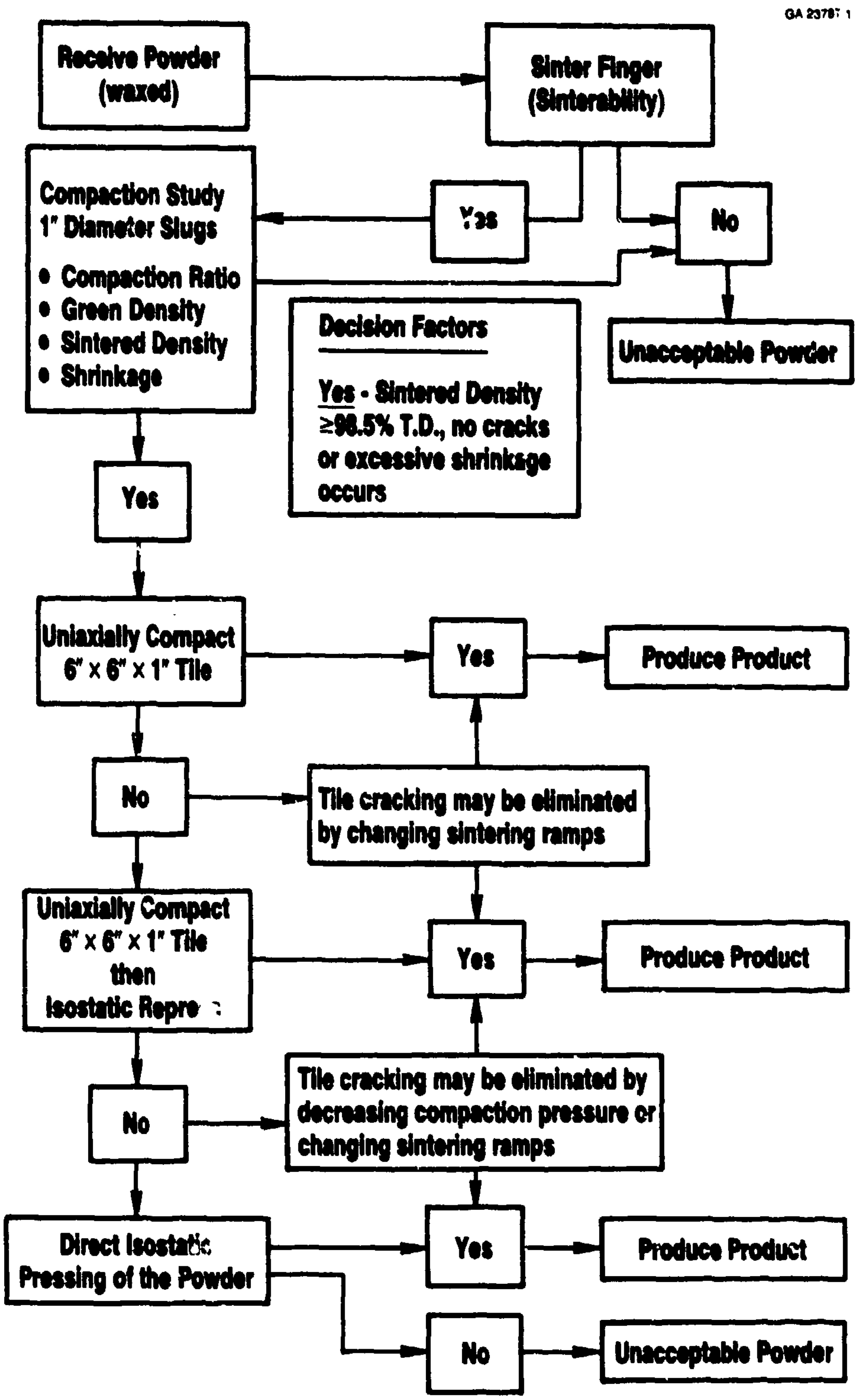
Typical microcrack

Overfired TiB_2 Microstructure Showing Microcracked Grains

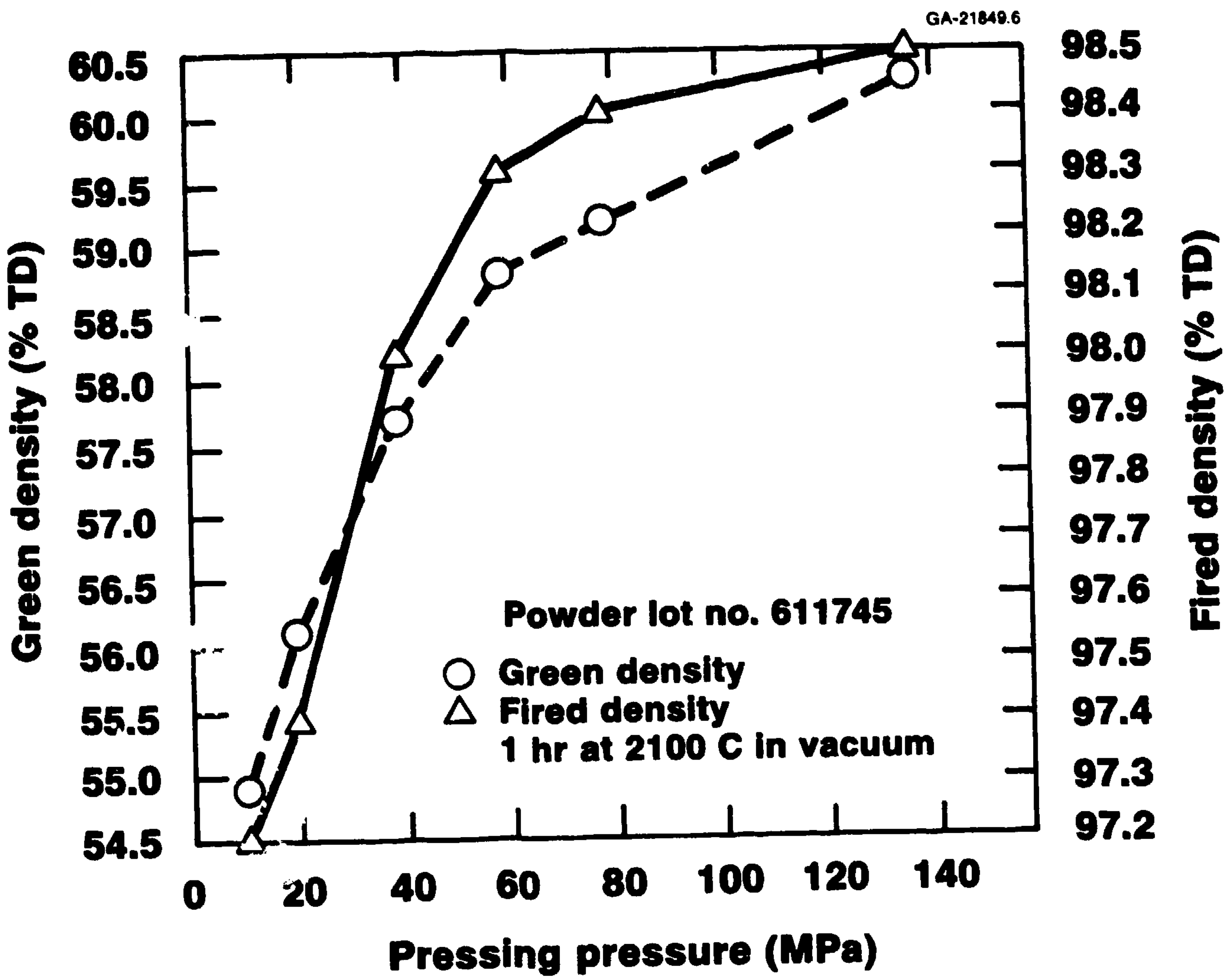


Microstructure of an Optimum TiB_2 Material. Black Spots are Attributed to a Carbide Phase

Comparison of Overfired and Optimum TiB_2 Microstructures
Figure 1

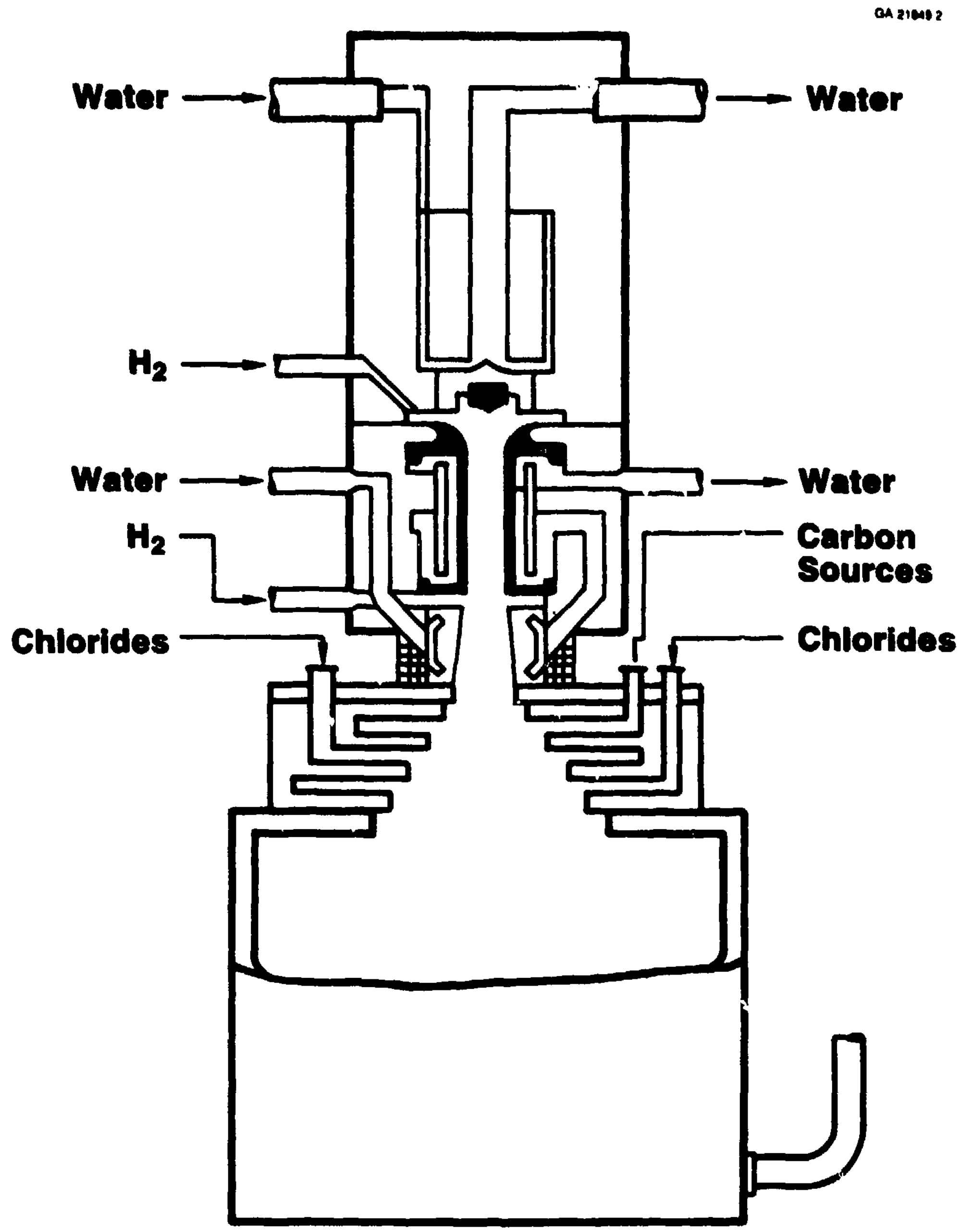


TiB_2 Powder Verification and Fabrication Development Flow Sheet
Figure 2



Green and Fired Densities of Processed Alcoa TiB_2
(Powder Lot No. 611745)

Figure 3



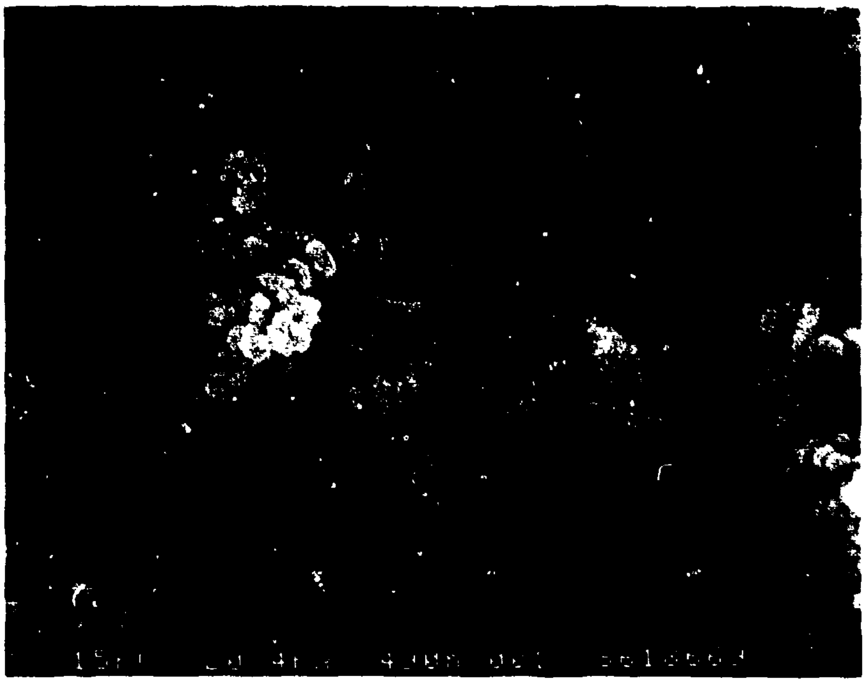
Reference U.S. Patent 4022 872

DC Plasma Reactor System

DC torch efficiency approaches 80% and for a range of operating conditions, the conversion of metal chlorides ranges from 50 to 90%. At the present operational scale, SiC can be produced at a rate of about 4.5 Kg/hr and TiB_2 at 7 Kg/hr.

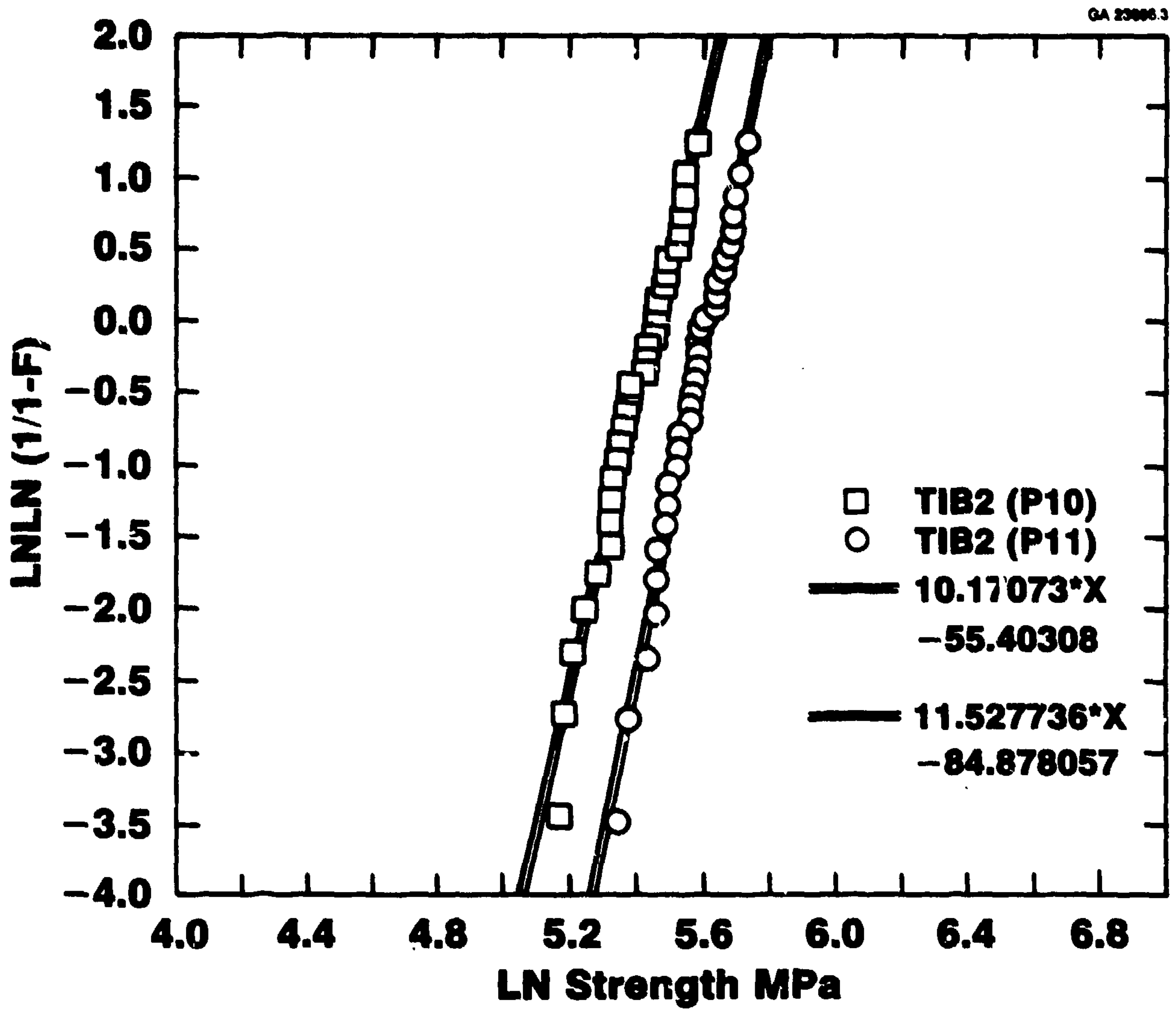
Plasma Powder Synthesis Equipment
Figure 4

GA 2366/8.1



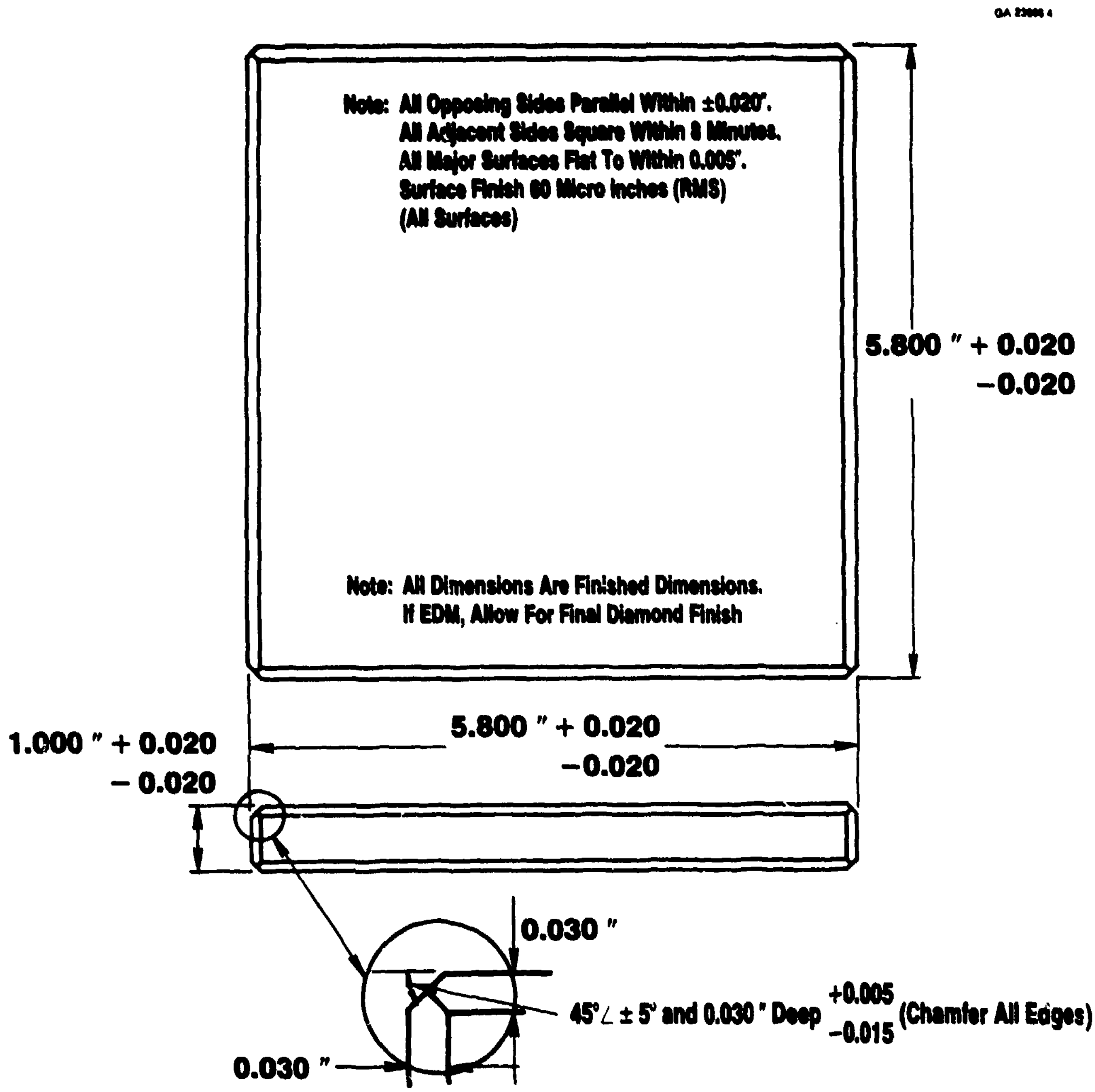
Alcoa Plasma T:B_2 Powder. Raw materials are T:Cl_4 and BCl_3 and a small amount of chlorinated organic to add carbon to the product. (~0.5 μm mass median diameter)

Figure 5



Titanium Diboride Weibull Plots

Figure 6



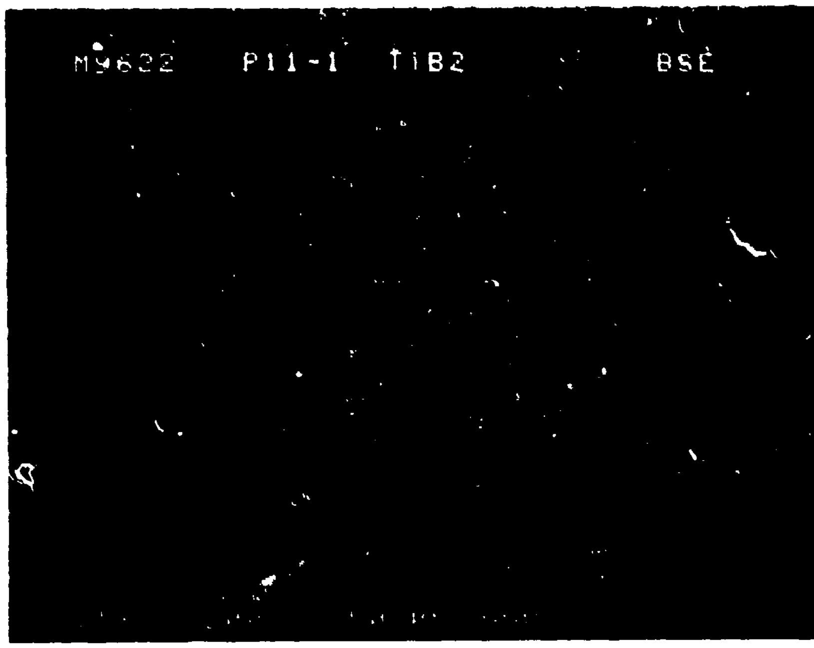
Dimensions for Alcoa TiB_2 Tile - MTL

Figure 7

GA 23886.6

Six - 5.8 × 5.8 × 1 Inch TiB_2 Armor Tiles Submitted to MTL for Ballistic Testing
Figure 8

GA 23608.5

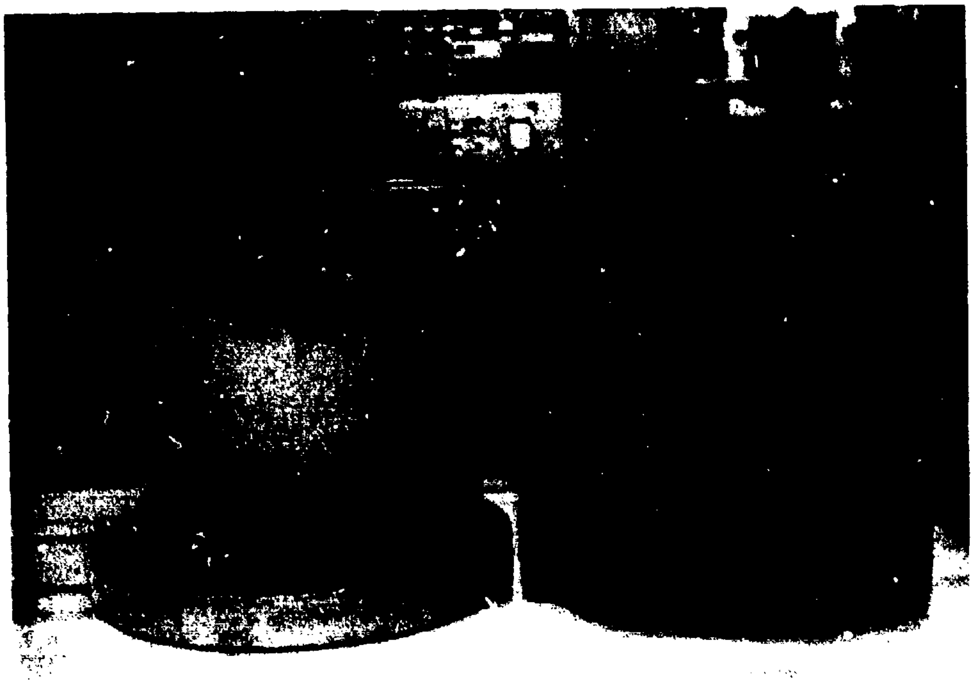


Low magnification photomicrograph showing void attributed to soft wax agglomerate (right hand side)

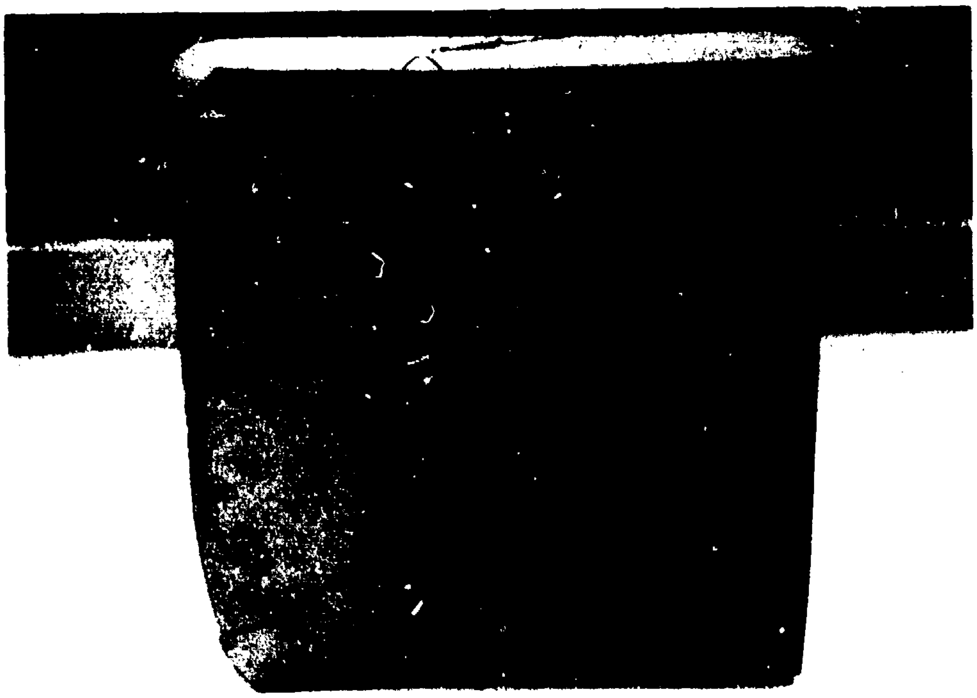
High magnification photomicrograph showing relatively microcrack - free grain structure - avg. grain size = 3.73 μm.

Photomicrographs of a Polished Section of a Specimen Taken from Tile P-11
Figure 9

GA 23696.7



Isostatic compaction components for fabricating the large TiB_2 tile



Large tile after sintering

9 × 9 × 3 - Inch TiB_2 Tile Fabrication
Figure 10

APPENDIX A

List of Technical Amendments to Contract DAAL04-86-C-0041

APPENDIX A

Technical Amendments - DAAL04-86-R-0041

Paragraph	Topic	Description of Amendment
Section C		P-0002* - All 6" x 6" x 1" tiles will be changed to 5.8" x 5.8" x 1.0"
C.2.2	Sintering Process Development (Phase I)	No change
C.2.2.1	Develop and Optimize Pressureless Sintering Process	P-0005 - "Best effort" try for one 9" x 9" x 3"
C2.2.2	Manufacture Plasma TiB_2 Powder for seven 6" x 6" x 1" and five 9" x 9" x 3" Tiles	P-0002 - Eight 5.8" x 5.8" x 1" tiles P-0005 - Six 5.8" x 5.8" x 1" tiles P-0005 - One 9" x 9" x 3" tile
C.2.2.3	Fabricate seven 6" x 6" x 1" Ballistic Quality Tiles	P-0002 - Eight 5.8" x 5.8" x 1" tiles
C.2.3	Material characterization on one 6" x 6" x 1" tile from C.2.2.3	No change
C.2.3.1	Flexure Test (MIL-STD 1942 [MR]-B)	No change
C.2.3.2	Flexure Strength (390 MPa ± 25 MPa)	P-0001 - Flexure strength range: 240-390 MPa
C.2.3.3	Calculate Weibull Modulus	No change
C.2.3.4	Measure density of the tile	No change
C.2.3.5	Measure Knoop microhardness (2000 kg/mm^2, 2000 gm load)	No change
C.2.3.6	Determine Young's Modulus (80 ± 5 x 10^6 psi)	No change
C.2.3.7	Conduct microstructural examination	No change
C.2.3.8	Six 6" x 6" x 1" deliverable to MTL (0001AA)	P-0002 - All sizes changed from 6" x 6" x 1" to 5.8" x 5.8" x 1"
C.2.3.9	Five (minimum) will be machined to 6" x 6" x 1" ± 0.020"	P-0002 - All tiles changed to 5.8" x 5.8" x 1"

*Contract Modification Number

APPENDIX A
Technical Amendments - DAAL04-86-R-0041

Paragraph	Topic	Description of Amendment
C.2.3.9.1	All opposing sides parallel within ± 0.020"	No change
C.2.3.9.2	All adjacent sides square to within an angle of eight minutes	No change
C.2.3.9.3	All major surfaces flat to within 0.005"	No change
C.2.3.9.4	Chamfer of 0.030" +0.005", -0.015" at an angle of 45° ± 5° from the major surface on all corners	No change
C.2.3.9.5	All surfaces finished to 0.060" (RMS - maximum)	No change
C.2.4	All data from C.2.3.1 through C.2.3.7 shall be delivered at the time of program review	Schedule dates changed
C.2.5	Program review ten months from contract initiation	Schedule dates changed
C.2.6	Review of program. Decision for proceeding to Phase II will be made by MTL	Schedule dates changed. Phase II not initiated
C.2.7	Funding go-ahead on Phase II by MTL	P-0005* - Deleted
C.2.8	Phase II Development	P-0005 - Deleted
C.2.8.1	Design, fabricate and install dies for producing 9" x 9" x 3" TiB_2 tiles	P-0005 - Deleted. Tooling for best effort furnished by Alcoa
C.2.8.2	Fabricate five ballistic quality 9" x 9" x 3" tiles with the properties described in C.2.3 using the processing conditions from Phase I. The powder produced in C.2.2.2 will be used for the 9" x 9" x 3" tiles	P-0005 - Decreased to one 9" x 9" x 3" "best effort"

*Contract Modification Number

APPENDIX A
Technical Amendments - DAAL04-86-R-0041

Paragraph	***Topic***	***Description of Amendment***
C.2.9	One 9" x 9" x 3" tile will be selected and characterization performed per C.2.3.1 through C.2.3.7. The other four tiles will be delivered to MTL	P-0005* - Deleted
C.2.10	The four tiles will be delivered to MTL within 14 months from start of Phase II	P-0005 - Deleted

*Contract Modification Number

APPENDIX B

Letter of Compliance
5.8" x 5.8" x 1.0" Tile Machining

Eagle-Picher Industries, Inc.

SPECIALTY MATERIALS DIVISION BORON DEPARTMENT

P. O. Box 798 Quapaw, Oklahoma 74363 918-673-2201

September 16,1987

Aluminum Company of America
Alcoa Technical Center
7th Street Road, Route 780
Alcoa Center, PA 15069

Reference: P.O. TC294045QA
TiB_2 tiles

Gentlemen:

This is to advise you that the six TiB_2 tile as provided by you have been ground and are being shipped to you today via UPS Blue.

These tile have been ground to the specifications of your revised drawing dated 87-08-13 with the exception of length and width which are nominally 5.953 which was authorized by your office to Jim Everitt and J. Boyer 8/24/87. Authorization provided that parts could be ground to minimum of 5.8 if necessary.

This letter serves as statement of conformance as per your purchase order request.

If there is any other information or assistance we may provide to you, feel free to contact us.

Sincerely,

Joyce Boyer

Joyce Boyer
Q. A. Manager

Attachment
to P.L. #1756

cc: Jim Everitt
EP Contracts

APPENDIX C

Linear Shrinkage Values for TiB_2 Tiles

APPENDIX C

Linear Shrinkage Values for TiB_2 Tiles

Tile	Dimensions (Inches) Before	After	% Shrinkage
P-10[1]	7.512 x 7.512 x 1.659	6.258 x 6.240 x 1.379	16.7 x 16.9 x 16.9
P-11[1]	7.510 x 7.511 x 1.700	6.166 x 6.151 x 1.415	17.9 x 18.1 x 16.8
P-12[1]	7.512 x 7.512 x 1.461	6.191 x 6.168 x 1.219	17.6 x 17.9 x 16.6
P-13[1]	7.512 x 7.512 x 1.438	6.171 x 6.174 x 1.201	17.9 x 17.8 x 16.5
P-14[1]	7.508 x 7.514 x 1.463	6.127 x 6.142 x 1.207	18.4 x 18.3 x 17.5
P-15 [1]	7.509 x 7.515 x 1.422	6.134 x 6.150 x 1.184	18.3 x 18.3 x 16.7
P-16[1]	7.509 x 7.512 x 1.440	6.132 x 6.150 x 1.204	16.3 x 15.7 x 16.4
P-17[1]	7.510 x 7.514 x 1.371	6.133 x 6.156 x 1.138	16.3 x 15.9 x 19.4
P-30[2]	7.516 x 7.516 x 1.094	5.986 x 5.967 x 0.900	20.4 x 20.6 x 17.7
9x9x3[3]	12.842 x 12.023 x 5.671	10.000 x 10.465 x 4.360	22.1 x 13.0 x 23.1

NOTES: [1]Powder Lot No. 611745, Low Surface Area Powder
[2]Powder Lot No. 618654, High Surface Area Powder
[3]Powder Lot Nos. RL-09-15 and RL-09-16, High Surface Area Powder

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U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
LOW-COST SINTERING OF TITANIUM DIBORIDE FOR ARMOR
M. W. Vance
Aluminum Company of America
Alcoa Technical Center
Alcoa Center, PA 15069
Technical Report MTL TR 88-27, October 1988, 49 pp.
Illus.-tables, Contract DAAL04-86-C-0041
Final Report, April 22, 1986 to August 31, 1988

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Key Words

Titanium diboride, Reactive gases, Powders, Sintering, Flexural strength, Young's Modulus, Hardness, Armor

The primary goal of this project was to fabricate lower cost pressureless sintered titanium diboride (TiB_2) armor tiles that would exhibit competitive ballistic performance with tiles formed by hot pressing. This was accomplished by using submicron titanium diboride powders synthesized by a plasma/gas phase reaction process originally developed by Pittsburgh Plate Glass Company. An initial task of Phase I of this effort was to develop and optimize fabrication processes for producing armor tile from plasma TiB_2 powders having various compaction and sintering characteristics. This task was followed by manufacturing sufficient quantities of TiB_2 powder to fabricate 6" x 6" x 1" tiles and a 9" x 9" x 3" tile. From the same lot of powder, eight 6" x 6" x 1" tiles (the machined tile size was later amended to 5.8" x 5.8" x 1") were produced and two of these tiles were used for characterization tests. The results of these tests determined that further tiles required isostatic repressing following initial uniaxial compaction to achieve acceptable properties. The remaining six tiles processed via repressing were delivered to MTL for ballistic tests completing the Phase I effort. Phase II was originally composed of fabricating five (5) 9" x 9" x 3" TiB_2 tiles. Although properties of the Phase I tiles were acceptable, the Phase II effort for producing the 9" x 9" x 3" tiles was not performed, and instead a "best-effort" attempt at manufacturing one large tile to determine process feasibility was implemented. Compaction of the TiB_2 powder into a green tile was accomplished. However, the tile developed excessive crack formation during the sintering process. This same crack formation had been arrested during the fabrication of 6" x 6" x 1" tiles by improving compaction and sintering processes. Also, processes for fabricating 6" x 6" x 1" tiles from high-surface area TiB_2 powders were successfully developed.

During this project, it was observed that to attain low-cost fabrication, machining pressureless sintered TiB_2 tiles to final dimensions should be minimized or eliminated by processing to net-shape dimensions. Non-machined 6" x 6" x 1" tiles were fabricated from the same powder lot to within 1/16" as part of Phase I. However, damage that occurred as a result of repressing required the tiles to be machined to 5.8" x 5.8" x 1.0". Extensive process development will be required to successfully fabricate 9" x 9" x 3" tiles. Additionally, a larger capacity inerted uniaxial press and a continuous charging vacuum furnace would be required to process large quantities of 9" x 9" x 3" tiles to achieve net-shape and low-cost sintering.

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